JPRS: 26,543

24 September 1964

TT: 64-41801

60536

RELATIONSHIPS BETWEEN TEMPERATURE AND DEFORMATION IN METALS

- USSR -

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RELATIONSHIPS BETWEEN TEMPERATURE AND DEFORMATION IN METALS

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Following is a translation of two articles in the Russian language periodical Izvostiya vysshikh uchebnykh zavedeniy, Fizika (News of the Higher Educational Institutes, Physics), No 2, Tomsk, 1964. Complete bibliographic information accompanies each article.

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SOURCES OF EXCESS VACANCIES WHICH FORM DURING SINTERING OF METAL POWDERS

[Following is a translation of an article by A. P. Savitskiy, V. I. Itin, V. N. Zhdanova, and Yu. I. Kozlov, Siberian Physics and Engineering Institute at Tomsk State University, in the Russian-language periodical <u>Izvestiya Vysshikh Uchebnykh Zavedeniy</u>, Fizika (News of the Higher Educational Institutes, Physics), No 2, 1964, Tomsk, pp 110-115.]

The influence of heating rate up to the isothermal holding temperature is investigated for copper powder briquettes which posses a different initial porosity on the magnitude of final shrinkage. The obtained results are in accordance with the diffusion diagram of sintering.

It is known that during sintering of powder metal materials rapid heating to the holding temperature can give greater compaction than slow heating /1--3/. Here, the fastest rate of shrinkage may not be at the actual temperature of sintering but in the process of its achievement. The given phenomen is explained by the fact that during rapid heating distortions of the crystal lattice are not eliminated in passing through low and medium temperatures and, therefore, must be removed in the region of high temperatures. Owing to supersaturation by vacancies, which form during correction of lattice defects, repair of micro-distortions, etc., the diffusion mobility of atoms during sintering turns out to be significantly increased which causes intense shrinkage.

In our opinion, this explanation encounters specific difficulties inasmuch as it does not agree with other firmly

established facts. For instance, after prolonged isothermal sintering, when shrinkage had almost stopped, further increase of temperature again sharply increases rate of shrinkage /4/. A similar regularity is observed at very high temperatures. To relate this effect to lattice defects and distortions is quite difficult since an overwhelming majority of defects disappear at comparatively low temperatures. The latter is so obviously brought to light in numerous experimental data on different types of crystal lattice defects and their temperature stability that one cannot discount its validity.

Furthermore, if to even allow that, in correction of the lattice, excess vacancies are formed, then it is difficult to grasp why there will be shrinkage as a result of this and not the formation of new pores owing to coalescence of vacancies and an even greater growth of old vacancies at the expense of vacancy escape, as is observed in other cases of

lattice supersaturation with vacancies /5--87.

It should also be noted that the considered explanation has not been confirmed by direct experimentation. Moreover, available data indicate that, for instance, dislocations do not render an influence on the process of shrinkage /97, and that deformation during pressing after pre-sintering, although leading to a change in density, but at the expense of creating lattice distortions by shrinkage during subsequent sintering, is not influenced /107.

The authors of this work hold the point of view, which, in their opinion, inevitably follows from the works of B. Ya. Pines /11/, Ya. Ye. Geguzin /12/ and other researchers, that theonly essential source of excess vacancies in a porous solid at high temperatures is the presence of the pores themselves. The diffusion mechanism of sintering porous crystal materials can be presented as vacancy dissolution of pores under conditions when the basic role in excess vacancy departure to the external surface of solids is related to grain boundaries and subboundaries /9, 13/.

As was first shown by B. Ya. Pines /ll/, the local concentration of vacancies ξ_r near a surface with radius of curvature r may be expressed by the magnitude of surface tension σ and temperature T in the following manner:

$$\xi_{\rm r} = \xi_{\rm o} + 26/r(2/kT)\xi_{\rm o} \tag{1}$$

where ξ_0 --equilibrium concentration of vacancies at temperature T, and Ω --atomic volume.

From equation (1) and general considerations it follows that the average concentration of vacancies g in a lattice containing vacancy sources depends not so much on the overall porosity of the material as to pore size and their

volume distribution. Obviously, the greater the number of pores per unit volume of material, the smaller the distance between them and the smaller their radius, the larger will be E.

The rate of pore radius decrease, due to dissolution, is proportional to the concentration gradient near a pore:

$$dr/dt = D_v(\partial \xi_r/\partial r) \approx D_v(\xi_r - \bar{\xi}/r)$$
 (2)

where Dv--coefficient of vacancy diffusion.

From (2) it is clear that for each supersaturation $\Delta \xi = \xi - \xi_0$ pores can exist in equilibrium with a solution of vacancies in the lattice $\bar{\xi}$. The radius of these pores, which can be called critical, is equal to:

$$\mathbf{r}_{cr} = (262/kT)(\xi_0/\Delta\xi) \tag{3}$$

At fixed temperature T the magnitude of the critical radius depends on the relative supersaturation of the lattice with vacancies $\Delta 5/5_0$: the smaller the supersaturation, the larger the pores of critical size. In a total absence of surplus vacancies in the lattice, pores of any size will be subcritical and have to be dissolved; conversely, a lattice containing pores should have some excess above the equilibrium concentration so long as pores will not be dissolved completely.

On the other hand, each temperature of sintering at a given relative supersaturation corresponds to a critical radius of pores, the size of which remains constant with time. Pores having a subcritical dimension have to be dissolved, emitting vacancies, while pores of supercritical dimension

must grow, absorbing vacancies.

The rate of shrinkage of a briquette obviously depends on the relative supersaturation, determining the drop in vacancy concentration near the external surface of a solid and in its volume and on the coefficient of vacancy diffusion.

Here, it is essential that supersaturation does not take place at the expense of some special sources in the actual lattice, but due to dissolution of pores. For the process of shrinkage it is also necessary that not the pores but the grain boundaries to emit vacancies to the surface. Otherwise, instead of shrinkage, only pore coalescence will take place.

In principle, the process of sintering should proceed at any temperature independent of pore size even if only the vacancies have some mobility. Pore sizes only determine the degree of supersaturation at which this process

is carried out. The sintering rate depends not so much on degree of superation as to the diffusion coefficient of vacancies. The latter is wholly determined by temperature and solid structure.

Thus, if one were to consider dissolution of a single pore, then relative local supersaturation $\Delta \mathcal{E}/\mathcal{E}_o$, as one may see from (1), should change proportionally with temperature as a result of which the rate of its dissolution should drop according to this law. However, the diffusion coefficient $D_{\mathbf{v}}$ increases with temperature exponentially, therefore, in the final analysis, the rate of pore dissolution should be sig-

nificantly increased with rising temporature.

Under actual conditions a pressed briquette contains pores of different sizes. The initial degree of lattice supersaturation at a given sintering temperature is deter- . mined by the relative amount and absolute size of the small-Shrinkage rate at the start of the process will est pores. depend on rate of vacancy departure to the surface of the A sharp drop in shrinkage rate during isothermal holding can be explained by the fact that according to the degree of dissolution of pores, due to the presence of a large number of sinks in the form of pores, of supercritical size, and grain boundaries, the degree of supersaturation decreases In order to sustain a constant rate of shrinkage it is necessary to maintain one level of supersaturation by measuring the corresponding drop of superation, or to increase the diffusion coefficient of vacancies. The first possibility turns out to be impossible since, during sintering, the process of pore redistribution, according to size, proceeds continuously decreasing the number of small pores and increasing the number of large ones. The second (increasing vacancy diffusion coefficient) is possible by simply increasing temperature.

Increasing temperature, according to (3), should give

Increasing temperature, according to (3), should give a negative effect on sintering inasmuch as there is a decrease of critical radius and, consequently, the number of dissolved pores. However, this decrease is compensated for by a growth of critical pore size owing to a decrease in degree of supersaturation of the lattice by vacancies due to an increase in

vacancy mobility.

From the above-stated one can conclude as to how it will qualitively effect heating rate during sintering, and also the effect of initial structure on magnitude of final

shrinkage.

Slow or stepwise sintering of a solid, containing pores of various sizes, should be unfavorable because the number of subcritical dissolved pores per time turns out to be very small, and all remaining pores serve as vacancy departure points, inasmuch as their size is more critical. As a result of pore growth of supercritical size in

the preceding stages of slow heating to temperature T_1 , with a subsequent increase in temperature to $T_2 = T_1 + \Delta T$, the number of dissolved pores turns out to be less, but growing larger as compared to the case when sintering is carried out from the very beginning at temperature T_2 . In other words, during very slow heating not so much shrinkage of the solid should be observed as coalescence of pores. Inasmuch as in this mode of sintering the number of effective sources of vacancies turns out to be small, and the average excess vacancy concentration in the lattice will be insignificant.

For departure of vacancies to the external surface of a solid the most favorable condition, as already stated, should be maximum degree of supersaturation. Such conditions have to be realized at high heating rates when pore redistribution according to size proceeds in the direction of decreasing number able to be dissolved at a given temperature, and an increase in the number of pores able to only grow.

The influence of powder size and their porosity for a given compact porosity should tell, in the first place, the average magnitude of interpartical pores and their quantity per unit volume and, consequently, the relationship of the number of subcritical and supercritical pores. Naturally, the smaller the particle size, the smaller will be the pores formed between them during pressing.

Particles with a smoother surface and the same size will give larger pores in contact than particles with an uneven surface. It is also obvious that porous particles have a reduced density but the pores contained inside them will form small pores between themselves which are smaller than the monolithic particles.

Consequently, preliminary annealing of powders, promoting smoothing of particle surfaces /147 and decreasing their porosity, should lead to compacts with larger pores. Subsequently, this will lead to a decrease in the magnitude of shrinkage during sintering.

It is easy to qualitatively trace this effect on the magnitude of final shrinkage and certain other factors. For instance, inasmuch as the coefficient of grain boundary diffusion is several orders of magnitude higher than the volume coefficient, then sintering of these compacts should be more effective than with finer initial powders.

The effect of oxides is probably ambiguous. On the one hand, although hampering interpartical recrystallization, oxides promote preservation of well-developed boundary networks in the sintered material which are ideal ways of emitting vacancies at the external surface of a solid. On the other hand, being decomposed at high temperatures, oxides inside closed pores, along with gases, can prevent dissolution

.of pores /157.

In the present work the task of an experimental check of conclusions about the influence of heating rate during sintering on the process of shrinkage was laid out, logically ensuing from the above-stated method of sintering.

In particular, it is possible beforehand to expect what will be the nature of pore distribution in samples at various heating rates up to the sintering temperature and an identical time of isothermal holding. First, samples obtained as a result of slow heating of compacts and holding at a high temperature must contain more and larger pores than samples produced under the same conditions but with rapid heating. Secondly, in view of a higher degree of average supersaturation of the lattice with vacancies at the time of reaching the sintering temperature, the size of the largest pores in the case of rapid heating should be significantly larger than the size of the largest pores in slowly heated samples.

A check of the above-mentioned conclusions was conducted on copper powder with particle sizes less than 50 microns. Cylindrical samples 15-20 mm long and 7 mm in diameter were prepared by bilateral pressing. In order to consider the possible effect of gas on the shrinkage process, high pressing pressures of varying intensity were used so that samples contained a different initial porosity prior to sintering.

Sintering was carried out in a vacuum (5 x 10⁻³ mm Hg) at 900 C for 1 hour. The average heating rate of one batch of samples was 1.5 degrees per minute, the other--approximately 200 deg/min.

After hydrostatic sintering the final porosity of the compacts was determined. Samples which contained the largest initial porosity were subjected to metallographic analysis. Results of these investigations are shown in Fig. 1.

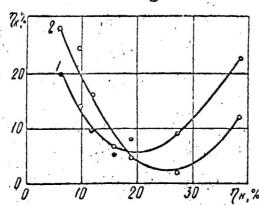


Fig. 1. Relationship of final porosity to initial for briquettes subjected to slow (1) and rapid (2) heating to the temperature of isothermal holding.

The general path of the curves in Fig. 1 agrees with the results of work /157. Furthermore, from this figure it follows that at a small magnitude of initial porosity, achieved by means of increased pressures, slow heating to the sintering temperature gives a smaller briquette expansion than rapid Obviously, this can be explained by the effect of heating. gases which remain in closed pores forming during pressing If, however, and gan be partially removed during slow heating. the effect of gases is not essential, which is characteristic for a high initial porosity, then fast heating turns out to be more effective.

Thus, experimental results of this work qualitively agree with the above-stated mechanism of diffusion sintering of compacts from crystal powders and is the basis for the idea of Ya. I. Frenkel on the leading role of vacancies in . this process, and B. Ya. Pines concerning increased concentration of v cancies near pores, and also numerous investigations of other authors on the kinetics of sintering. mechanism makes it possible to explain, not resorting to special assumptions about the existence of some specially stable defects in the lattice at high temperatures which create a concentration of vacancies, why the heating rate renders more effect on the density during sintering than total sintering time.

At present it is possible, with sufficient foundation, to affirm that in metal powders, sintered at temperatures close to their melting point, even if there are sources of excess vacancies then there can only be pores. With regards to grain boundaries, a well developed network can be preserved in metal powders a longer time at sufficiently high temperatures, while in the presence of pores, these powders are a very effective sink, emitting excess vacancies to the external surface of the sintered body.

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CSO: 1879-D/PE

ROLE OF VISCOUS DISLOCATION MOTION

TEMPERATURE-STRAIN RELATIONSHIP OF SUBSTITUTIONAL SOLID SOLUTIONS

Following is a translation of an article by L. Ye. Popov, L. M. Butkevich, and N. A. Aleksandrov, Siberian Physics and Engineering Institute at Tomsk State University, in the Russian-language periodical Izvestiya Vysshikh Uchebnykh Zavedeniy, Fizika (News of the Higher Educational Institutes, Physics), No 2, 1964, Tomsk, pp 126-130.

I. The temperature dependency of the characteristics of deformation resistance of many substitutional solid solutions clearly reveals a pronounced break in the temperature component at ordinarily applied rates of deformation (10^{-3} - 10^{-4} sec) approximately 0.5--0.6 of the melting point. Above this temperature (T_{max}) the characteristic of deformation resistance drops rapidly 2^{-6} . In many cases T_{max} precedes an anomalous temperature dependency of deformation resistance 2^{-4} , 6, 2^{-6} .

In Fig. 1 a, b, c, d, examples of the temperature dependency of different mechanical properties are given for mono- and polycrystals of a number of alloys. Breaks, corresponding to T_{max} , are indicated by arrows.

By analyzing data similar to that in Fig. 1 (for instance, see the detailed survey of Westbrook /1/), it is possible to note the following basic features of the considered phenomenon.

l. The sharp drop of mechanical properties at temperatures above T_{max} is observed for alloys with different crystal lattices both in stoichiometrical (ordered) composition

(Cu₃Au, 6-brass, CoFe, Ni₃Fe, Pt₇Cu /17, and also in which there is only short-range order (~-brass, Ni+20%Cr /5, 77, and aluminum bronze/37).

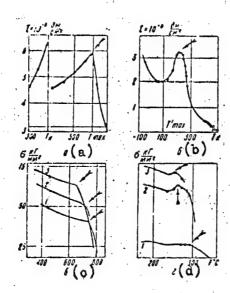


Fig. 1. Relationships of temperature to mechanical properties of various alloys: a--yield strength of Cu₃Au single crystal; b--yield strength of C-brass single crystal; c--flow stresses in Ni+28%Cr alloy at 6, 12 and 18% deformation (curves 1, 2, and 3 respectively); d--yield strength (curve 1) and flow stresses at 12 and 18% strain (curves 2 and 3) for L62 <-brass.

- 2. In ordered alloys of stoichiometrical composition (or close to it) $T_{\rm max}$ may be both below (Q-brass /4/, CoFe /1/, (Ni, Fe)_3Al /8/), and also above (Cu_3Au, Ni_3Fe, Pt_7Cu/1/) the temperature of disordering $T_{\rm k}$.
- 3. A sharp break of the temperature dependency of mechanical resistance occurs both for mono- and for polycrystals 2, 4, 7, 87.
- 4. Temperature T_{max} is increased with growth of strain rate and drops with increase of degree of strain. The latter is well illustrated in Fig. 1, c and d, where graphs of the temperature dependency of flow stresses for κ -brass and an alloy of nickel with 28% chromium for various degrees of strain are given.

On the basis of the above statements it is possible to

express the following considerations.

l. For substitutional solid solutions of varying nature it is apparent that one should not look for (in each case) special mechanisms in the rapid drop of mechanical properties at temperatures above the temperature T_{max} . On the basis of the change in the temperature path with respect to deformation resistance of solid solutions, similar changes of the mechanism of deformation probably exist.

2. The phenomenon cannot be explained by only viscous behavior of grain boundaries at high temperatures, inas-

much as the effect occurs also in single crystals.

II. Flynn /87 expressed the assumption that the sharp drop in the proportional limit of an ordered alloy (Ni, Fe)3Al, and also -brass, at temperatures far below the disordering temperature is caused by a build-up in the rate of "viscous" motion of dislocations joined in pairs by bands of anti-phase boundaries and not having a general plane of slip.

Under the viscous motion of dislocations, in the case of motion of dislocation pairs, they move with the help of thermal variations together with anti-phase boundaries between them.

The idea of viscous dislocation motion was also used to explain the creep of alloys with short- and long-range order /1, 97, and also to explain the upper temperature boundary and phenomenon of intermittent strain in case of blocking dislocations by Suzuki /10, 117. The movement of dislocations independent of the braking mechanism is called viscous if: a) it is accompolished with the aid of thermal fluctuations and, b) the distrubed braking atomic configurations are restored (by diffusion means) directly in the course of dislocation shift.

Owing to thermal fluctuations, viscous motion of dislocations can be carried out at stresses lower than the stress necessary for starting athermic motion (for instance, with the breaking away of atmospheres from the dissolved atoms which destroys the close-range order or forms an anti-phase boundary in the slip plane of ordered alloys).

On the basis of the expressed considerations it is possible to explain the change of the temperature dependency of deformation resistance at T_{max} in the following manner. With an increase of the temperature the contribution of viscous motion of dislocations in strain increases. In the region of temperatures, at which the speed of viscous dislocation motion is insufficient, in order to ensure rate of deformation, externally applied, it is necessary for an athermic motion of dislocations to participate in the deformation. In this region of temperatures there is a weak (or anomalous)

temperature dependency of mechanical properties. A strong temperature dependency should exist at those temperatures where deformation is carried out only by means of dislocation motion (viscous).

Thus, it is possible to consider T_{max} as the temperature above which deformation provides a completely viscous motion of dislocations.

For extimating T_{max} we will take that rate of deformation at a temperature approximately equal to the rate of micro-creep under the same conditions /12, 137.

For rate of shear deformation during micro-creep & independent of the mechanism of viscous dislocation motion, Weertman/13/ found the following expression:

$$\approx (ANb)\tau^2 b^2L/6AB$$
 (1)

where A--lenth of slipping dislocation, N--number of dislocation sources per unit volume, τ --tangential stress in slip plane, L--distance of dislocation threshold from source to obstacle, B = $Gb^2/2\pi(l-\gamma)$, and A--a coefficient determined by the relationship:

$$v = \Delta \tau b/A$$
 (2)

Here ν --rate of viscous dislocation motion, and $\Delta \tau$ --effective stress for moving dislocations.

An exact calculation of rate of viscous dislocation motion is apparently quite complicated. However, it is possible to estimate the limiting value of this rate v_k by the method used by Shock /14/ for the case of short-range solid solutions. For the maximum speed, the maximum rate was taken at which short-range order, destroyed by dislocation slip, succeeds to be restored in time of dislocation advance to a distance on the order of its Burgers vector b. According to /14/, the time should be on the order of the time for probably jump of an atom, i. e.;

where G--number of atomic jumps per second.

Inasmuch as

$$D \approx a^2 v \exp(-Q/RT) = a^2G,$$

where D--diffusion coefficient, and Q--activation energy, then

$$v_k = b/\tau \approx Gb \approx Db/a^2 = D/2b$$
 (3)

The given calculation is applicable to all the abovementioned mechanisms of dislocation braking. Combining (2) and (3), we find

$A \approx 2b^2 \Delta \tau / D$

Assuming $\Delta \tau$ equal to stress $\Delta \tau_k$, necessary for the start of an athermic dislocation motion, also considering that $\Lambda \approx 3L$ and N $\approx 1/L^2h$ (h--distance between slip traces), we obtain

$$\gamma \approx \kappa(\tau^2/\Delta \tau_k h) D$$
 (4)

If for the rate of viscous dislocation motion we take expression (1), then K=3/8 (1- ν /Gb²).

In the case if data relative to h are absent, then for the rate of micro-creep it is possible to use Weertman's formula /127:

$$\gamma \approx (\tau^3 b^2/GAB)$$
 (5)

Finding T_{max} according to formulas (4) and (5) were previously done by us for L62 &-brass and Nichrome with 28.5% Cr /117. The obtained values are presented in the Table. Here values of T_{max} are given for Cu₃Au and 8-brass found from /57.

TABLE

Сплав	Коэффици- епт диффу- зии <i>D</i> при b) T _{max}	Коэффициецт диффузии (о)	Источ (р)	Скорость деформации, <i>сек</i> —1	(f) Т _{тах} (теор.)	(g) Т _{тах} (эксп.)	T _{max} T _{nh}
Ni+28,5% Cr	0.8.10-10	$16^3 \exp\left(-\frac{85000}{RT}\right)$	[4]	1,2.10-4	810	780	0,63
(і) Латунь Л62	2,3·10 ⁻⁹ £	$0.73 \exp\left(-\frac{34000}{RT}\right)$	[11]	1,2.10-4	310	290	0,45
Cu₃Au	6.10-10 6	$0.1 \exp\left(-\frac{44000}{RT}\right)$	[15]	4,4.10-4	570	600	0,68
(ј) 8-латунь	5·10 ⁻¹⁰ s	$77 \exp\left(-\frac{35600}{RT}\right)$	[16	$ 10^{-5} - 10^{-2}$	200- -260	200	0.45— —0.50

Key: a--alloy; b--coefficient of diffusion D at T_{max}; c--coefficient of diffusion; d--source; e--rate of deformation, sec-1; f--(theoretical); g--exsperimental; h--T_{m.p.}; i--L62 brass; j-- -brass. In calculating T_{max} for yield point $\tau=5 \times 10^8$ dyne/cm² was taken for Cu_3Au /2/and $\tau=3.4 \times 10^8$ dyne/cm² for ε -brass /47. With reference to the yield point one can use $\Delta\tau_k \approx \tau$.

In view of the lack of data on parameters of diffusion of Cu and Au atoms in Cu₃Au, values of D and Q are taken according to Vineyard /15%. Parameters of diffusion of Zn and Cu atoms in Q-brass are taken from work /16%. Diffusion coefficients of atoms of an ordered phase are close to one another. The value of T_{max} for Q-brass, presented in the table, were obtained by substitution of the parameters of Zn diffusion in this alloy (D=77 cm²/sec and Q=35,000 cal/mole) into (4). Unfortunately, the rate of deformation was not shown in /4%, therefore, its most often used magnitude was taken:

10-5<8 < 10-2

It is evident from the table that values of T_{max} , calculated for deformation rates on the order of 10-4 sec-1, are for all the considered alloys in good agreement with magnitudes of T_{max} observed experimentally. (

III. As noted above, T_{max} drops with increased degree

of strain. This can be seen from Fig. 1 (c, d) and Fig. 2. Lowering T_{max} with growth of strain rate follows from equation (4). The strain rate, controlled by viscous dislocation motion, depends on the diffusion coefficient D and the factor τ^2/h . With increased strain, τ^2/h grows and the strain rate, externally applied, is provided at a smaller D and, consequently, at lower temperatures.

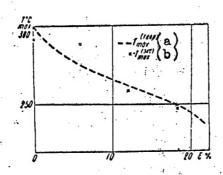


Fig. 2. Relationship of T_{max} to degree of deformation for polycrystalline x-brass: a--(theoretical); b--(experimental).

The physical meaning of the relationship of Tmax(€)is

simple: a given rate of deformation is provided where the smaller the velocity of viscous dislocation motion the greater

will be the length of moving dislocations, and, subsequently, the higher the dislocation density.

The dotted line in Fig. 2. shows the relationship of temperature vs. strain for polycrystalline L62 brass, which was calculated by us using equation (4). For polycrystalline

samples, equation (40 was transformed to the form:

$$\epsilon \approx \frac{3}{16} \frac{1-\nu}{\text{mGb}^2 \Delta o_{k}} \frac{\sigma^2}{h} D$$
 (6)

where \mathcal{E} --strain rate, $\Delta \phi_{k} = m \Delta \tau_{k}$, $m \approx 3.1$ --Taylor's constant,

and o--applied stress.

In the calculation there were used electron microscope data of Swann and Nutting //17/ on the change of distance between slip traces h in ≪-brass (30:70) in relation to the degree of strain. Values of of for various degrees of strain were determined from flow curves of L62 brass. Results of the calculation satisfactorily agreed with experimental values of Tmax for various degrees of deformation.

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